Notebook Series -- Notebooks by Experimenters Other Than Edison
Battery Experiments -- Experimenters' Books
E. E. Dougherty Notebook, N-19-08-25

This notebook was used by E. E. Dougherty during August 1919-January 1920. The entries pertain to the analysis and preparation of various chemical compounds for storage batteries. Some of the experiments relate to attempts to recover usable chemicals from the battery manufacturing process. Other entries pertain to checks of samples of nickle flake and electrolyte from the manufacturing process. Dougherty's numerous references to Edison, as well as several notes by Edison inserted into the book, indicate a close collaboration between the two men on this work. The pages are unnumbered, and the book has been used in both directions. Approximately 125 pages have been used.

Precipitate

Job No. 300 E.T. 209 Ni+Cu Carb

300-X-1VV Iron Oxide

C 700-X-V C.P. $NiSO_4$

C 300-X-4 Alkali Recovery

10/10/14

Green plastic bar
i Stag Flashing tank
3 Gas Flashing tank

Hamed in
Quantum jar
Ulc.

Aug. 25/19

Sample of Nickel-Copper Precipitate

Appearance :-

Green Hydrated Carbonate. —
{ Does not break up
{ readily with Cold Water.

Breaks up readily in Water & Pastle

Reaction (Reddening) paper :- Slightly alkaline

After boiling pulverized material in water :—

Filtrate :- Clear - Slightly alkaline.

Water Sol. Salts :- Not much in great deal
and principally Na_2SO_4 + NaCl
and free of metallic salts $(NH_4)_2S$ - Trace
contains $CaSO_4$ in solution

Even with xs Acid and boiling, lumps
do not break up readily.

Note Make a slurry in this Mind
at the outset, for, as the matter goes
into solⁿ it seems to cause an
occlusion.

There is considerable insol. —

Matter :— Metallic Nickel, Iron Oxide
Sand etc.

CJ

Aug. 25/19

1st Preparation

Raw Material :- Impure Nickel - Copper Precipitate, containing Iron - Metallic Nickel, Dirt, and Na_2CO_3 and Nickel & $CaCO_3$ etc.

Object :- To produce pure Copper Sulphate and Nickel Sulphate

Method

Add dilute H_2SO_4 - Dissolve all of the hydrate Carbonate possible - To insure neutral state add an excess in slight excess of above Carbonate.

Note :- In this experiment Fe did not go into solution. -

Filter - Wash slightly acid with H_2SO_4 - Precipitate Cu with H_2S - Wash CuS free of sol. salts - Roy - Give sulphatizing roast forming $CuSO_4$ which is leached away - Fresh is added to fresh batch of CuS

This will remove Ni and Fe from $CuSO_4$

Filtrate is concentrated to proper point for Mushing-out -

Caution. Mr. Edwin Mirick Power of H_2S

Meal is redissolved and re-crystallized - M-Liquors are used for dissolving new batch of precipitate.

The insoluble which is left from first dissolving operation, and which contains metallic Nickel particles is allowed to accumulate, when it is treated with strong sulphuric acid and slight additions from time to time of HNO_3 to insure solution.

Insoluble matter from this operation must be free of Nickel and Copper before being discarded.

Note

This process works out satisfactorily, but will take a great deal of H_2S, although this H_2S helps to form the final $CuSO_4$.

H_2S changing operation added to similar to Freiberg System.

Sept 5

Aug. 26th

2nd Proposition

Raw material and Object same as preceeding

Method

Add slightly less than the calculated amount of H_2S or necessary for combination with Nickel Carbonate — Very same as Acid Phosphate — Heat slightly if necessary. — Decant and examine for Copper.

The little trace of Cu left to be removed by H_2S. Ni(OH) or metallic Iron.

This is worth looking into as it gave a pretty good looking Chromic Oxide, and it may be more valuable as such, than as a source of Blue Vitriol.

Grind in presence of excess (slight) of H_2S or S free from Ni. — Heat collect and dry. Antifouling Paint

Aug 26th Look into this further

3rd Proposition

(Mr. Edison's Suggestion)

Make Neutral Sol. as in Exp. 1 — add an emulsion of finely divided precipitate. — Boil if necessary — (Nickel Carbonate should ppt. Cu from Sol.)

Took equal amounts of Precipitate. To the first added enough H_2SO_4 to dissolve the Carbonate of Nickel & Copper, and leaving a Neutral Sol. (Mr Edison) — To the second add enough H_2SO_4 from a thin slurry. — Add to the first sol with stirring. —

After standing at room temp. for 1/2 hr with occasional stirring a portion was filtered — Did not filter readily — Filtrate was slightly cloudy — Upon boiling, the solution cleared up with ppton of Nickel Hydrate — This sol. showed qualitatively more Nickel and less Copper than the original sol, but reaction was not complete (Showed no Fe in sol.)

(5)

Aug. 26th

3rd Proposition (Contin.)

Another portion of this mixture was boiled for about 10 min. — Showed a tendency to foam and froth, with evolution of CO_2. — Mixture took on darker colour. Filtered very readily, also washed easily — Filtrate almost water white, contained no Fe — or Cu — Did not contain as much Ni and Sulphuric as it should — ~~Too much Fe~~ Heating seems to have formed a basic, water insol. compound of the Sulphuric, Nickel and Copper.

On a large scale, this may be obviated by chem. control, and addition of further quantity of dil. H_2SO_4.

This exper. if carried out on a larger scale and controlled by analytical data, would undoubtedly give more satisfactory results. Must find out composition of the Ni-Cu H_2SO_4 compound more in H_2O

Aug. 26th, 9 p.m. note made before this portion began precipitating

Exp. 3 a

This belongs to Exp. No 3.

Took sol. of neutral $CuSO_4$ in test-tube & added powdered Cu-Ni ppt. in excess — Boiled — Nickel went into sol, and Cu ppted. — Conversion was far from complete. — Seemed to need a very large excess of Nickel ppt.

This expers. should be controlled by analytical data.

CO_2 was evolved.

Seems promising, but ~~not~~ as though the reaction was reversible.

H_2S seems to be the best ppting agent.

9/4/19

This has been okd by Mr Edison & he wants it tried out on large scale

Exp. #4

Dissolved the Ni-Cu ppt in H_2SO_4 forming neutral sol. — Form strong neutral sol. — Insure freedom from Fe by leaving ~~[illegible]~~ excess of original material. —— Filter — Retain insol. for further treatment.

Filtrate consists of $CuSO_4$ mostly with some $NiSO_4$ — Concentrate to smoking point. — Pass into Oliver or other suitable filter — Filtrate containing most of the $NiSO_4$ is passed to Tank #2 — Wash B.V. with sat. sol. of B.V. — This may be done by spraying the cake on filter, or by removing the dry cake from filter to a separate tank, where it is agitated with a sat. sol. of Potas Nitrate. — This wash sol. is used over and over again, (having its own tank) until $NiSO_4$ contents become too high when it is either transferred to Tank #2 or Tank #1.

Redissolve and recrystallize

<u>Contin.</u>

B.V. mass, to form Ni free $CuSO_4$ solution. — M-Liq. from this recrystallization may be used in washing B.V. mass of subsequent batches.

Copper Carbonate may be used for making B.V. wash-liquor, or may be dissolved in Tank #1, as found most convenient.

The M-Liq. from B.V. making operation (consisting of $NiSO_4$ principally, with considerable $CuSO_4$) has been transferred to Tank #2 where it is treated and Nickel Carbonate containing also some Copper Carbonate & Hydrate is added, until a drop touched to $K_3Fe(CN)_6$ paper shows absence of $CuSO_4$ — Before filtering see that sol. is as strong as possible, and that an excess of Nickel Carbonate is in suspension (this insures absence of Fe_2SO_4 and $CuSO_4$) — Filtrate should

Contin.

consist of pure Nickel Sulphate, which may be used as such or crystallized — If crystals are made, the M-liq. is to be concentrated for another batch of xtals, or used to best advantage.

In the preparation of the above Nickel Sulphate at Tank #7, insoluble Copper Hydrate will accumulate — This may be dissolved by H_2SO_4 in a separate Tank — filtered, and added to the Neutralizing operation, or it may be used for washing the B.V. Mud, or it may be added to Tank #1 as seems best.

Nickel flake and other metallic will be accumulated in time, at Tank #1, where it is dissolved in another room in other acid-proof apparatus, by a mixture of Sulphuric and the amount quantity provided of HNO_3 — Filter — Filtrate goes to Tank #1 or H_2 and the insoluble is discarded as valueless. —

Contin.

The whole process, may be manipulated in such a way, that the Iron accumulates at the operation at Tank #1 — When it is desirable to eliminate it from the process, simply add Ni + Cu ppt. Nickel Carbonate or Copper Carbonate to the solution high in Fe, until a drop, added to $K_3Fe(CN)_6$ test paper shows no blue spot. — This operation should be carried out at boiling temp. — Now bring back gradually by addition of dilute H_2SO_4, to point where a drop added to $K_3Fe(CN)_6$ paper, shows a decided blue spot. — Filter — Wash well — Allow filtrate and washings to go to Tank #1, and dis. and the process cease. (Oxidation by means of the HNO_3, etc and precipitation by means of $BaCO_3$ might be used at times.) — The concentration for $NiSO_4$ xtals, $CaSO_4$ may separate out, separate by settling or filtration. — In this process only H_2SO_4 and a very small amt of HNO_3 are used.

(11)

Spec. of Dilute H_2SO_4 from Mr Dimroldie
P.M. 9/10

23.3422
14.6933
99 = 17.4 cc H_2SO_4 taken m/2
= H_2SO_4 :- 50.7 %

24.4949
13.0505
11.4444 = 55.0 cc H_2SO_4 m/2
✓ = H_2SO_4 :- 12.11 %

Dilute Acid from Mr Dimroldie A.M. 9/13/04
24.5205
14.6925
9.8280 = 19.35 cc m/2 H_2SO_4
✓ = H_2SO_4 4.96 %

Dilute Acid from Mr Dimroldie A.M. 9/15/04
20.6520
14.6905
5.9615 = 16.2 cc m/2 H_2SO_4
✓ H_2SO_4 11.07 %

I find that H_2SO_4 necessary to dissolve original Mixed Carbonate (Rough)
11.65 gm = 98.0 cc m/2 H_2SO_4
∴ Result that 1 # Matte = 0.147 # H_2SO_4
or 0.15 # 93 % Sulph. Acid

Lots per cuft of Matte used for following Exp.

Curtis Matte from Pille

10 # 12 1/2
4 # 2 1/2 g. Jar Battery jar
6 # 10 oz. Nest

Water

14 6
4 # 2 1/2 g. Jar
10 # 3 1/2 g.
10 # 11 oz.

Nickel Carbonate

4 2 1/2
6 # 8 1/2
9 # 14 g.

Copper Carbonate

4 2 1/2 oz.
5 # 11 1/2

Therefore 1 cu ft

= Water	62.4 lbs
Ni + Cu Carbonate	40.48 "
Ni Carbonate	39.87 "
Cu Carbonate	34.92 "
67.66	114.47 "

Sept. 16th, 1918

Large Scale Trial of Cu ppt. #4

Crock #1	21" x 16" = 5.21	cu ft x 1" =	cu ft
" 2	21 x 16" = 5.21	" "	"
" 3	21½" x 16" = 5.46	" "	"

Crock #1 Dissolving Tank
" 2 Cu ppting "
" 3 Ni So4 Sol. Storage Tank

Cu + Ni Ppt. = grams per
= # per cu ft.

Cu + Ni Nitrate		H_2SO_4	H_2O
70#	7 oz.	13# 13½ oz.	15 litre
24 +	1¼ oz.		1 " for Washing
Total 94 #	8¼ oz.		16

Contin.

1st Operation. Added 70# 7 oz. Material + 15 litre H_2O — Could just about mix with a paddle, was very thick. — Added 13# 13½ oz. Sulph. Acid — Temp. rose to 50 °C. and did not show much tendency to boil over. — Sol. was strongly acid, therefore added remainder of Cu-Ni Nitrate = 24# 1½ oz. making total addition 94# 8½ oz. — Heated to 86 °C and showed 1.16 Spec. — Solin complete in 15 min. — Film of Scum & grease on top. — Solin neutral but contained considerable Cu in sol., ~~[illegible]~~ Insoluble and Cupric Hydrate in suspension settled readily — Seemed to be granular. —

Depth in tank and Spec. just before filtering = 8" and 1.182 @ 90 °C. Neutralized with some Cu So4 in sol.

Filter

Filtered quite readily, but a very small quantity of suspended matter escaped into the filtrate. — Filtrate transferred to Tank #2

Contin.

#1 Filtrate 3 r. litre × 320 cc
Took 300 cc for Spec

#2 Press Cake 23# 12 oz.
Took 385 grms. for Spec
Lost 100 " by Manipulating

2nd Operation

To the 3 r @ 320 cc Filtrate in Jump #7 added 6# 8 oz. of Nickel Carbonate & heated to boiling — Was not enough, showed Cu reaction by K_4FeCy_6 — Added 6# 4 oz. additional Nickel Carbonate, did not show Cu by K_4FeCy_6, but upon filtering and testing filtrate with H_2S a black ppt. formed. — Added finally 6# 3 oz. Nickel Carbonate Note Even after this last addition H_2S formed a ppt. — This black ppt. upon closer examination proved to be Nickel with very fainter tint of Cu (probably 1 part to 50000 parts of Nickel. — More Nickel Carbonate was added in this operation than was actually necessary, due to the black ppt showing — In subsequent

Contin.

work, be careful to add only what is actually necessary to ppt. all of the Copper. — In heating the mixture during this operation some concentration in Volume took place, — Had to add extra Water before filtering —

Filtered — Should have used Coarse Filter cloth but had none. — What was used being too close gave considerable trouble — Filtrate clear and free of Fe and Cu. —

Ordinarily this Cake is supposed to be added to Copper Carbonate for production of Br. V., but as proportion used was too great I knew it held too much Ni for this purpose, thought I would extract some of it which of course gave an extra solution, and lead press cake for analysis.

2nd Filtrate 20½ litres 20° Be
Took 2 litres for Spec
" 1 " " Cu & Fe test

Found it to be free of both Cu & Fe

Contin.

2nd Press Cake. Did not weigh, and expected to extract more Nickel from it producing another solution also another press cake which would be nearer the composition we would strive to obtain on a large scale. — Therefore mixed thoroughly in another vessel and added 200 cc H_2SO_4 — Filtered — Had some trouble as at first, due to plugging of the close woven cloth. — Had to remove from filter while still holding considerable solution

2.b Filtrate: 1470 cc (bottled all)

2b Precipitate very wet 23# 1 oz. Took 150 gm in handling

Set aside in large Porcelain Dish

Kept Filtrate in large Bottle

Contin.

3rd Operation

Dissolved Press Cake from #1 Tank to make Blue Vitriol

Took 23# 1 oz. minus 485 gm + 4# 8 oz Sulph. Acid in large porcelain Dish — Too acid so added 5# 11½ oz more Copper Carbonate — Heated to boiling — Filtered but did not wash very much — Filtrate 11 liter + 300 cc for Spec + 370 cc + 60 cc lost

Insol. 3# 7 oz.

Bottled the Insol.
Boiled down Filtrate in large Porcelain Dish

4th Operation

B.V. Wash Solution made by dissolving 15# 4 oz. Copper Carbonate with 470 cc H_2SO_4 (2nd lot)

Remark:—

Contin.

B.Y. Work solution rather diluted — only 119 lbs. at room temp. — concentrated — Filtered — Worked but not completely — Residue

Filtrate

Press Cake

The above sol. settles readily in cold, better still hot, but as a scum formed on top I poured all through filter. This sol. may contain nickel Flos.

Contin.

Contin.

1) Spec of 100# Mixed Ni & Cu Carbonate from Pile

25.414	25.414
12.345	14.737

Moist:-

Took 8.61 grms.

1/1 = 0.017 grm Insol.:-
= Insol. %

1/5 = 2.3 as $K_2Cr_2O_7$
= Fe

1/5 = 0.85 - 00 as Hypo
= Cu

1/5 = 12.2 - 13.7 as KCy
= 0.3 - 0.5 as $AgNO_3$
Ni

2) Spec of 50# Nickel Carbonate

18.352	18.352
9.210	11.058

Moist:-

Took 13.099 grm

1/1 = 0.043 grm Insol
= Insol.

1/5 = 5.7 as $K_2Cr_2O_7$
= Fe

1/10 = 2.7 - 00 as $Na_2S_2O_3$
= Cu

1/10 = 14.2 - 15.1 as KCy
06 2.6 as $AgNO_3$
= Ni

3) Spec of 50# Copper Cast Iron

22.265	22.265
11.225	12.5340
11.039	9.731

Moist 88.15

Took 11.890

1/1 = 0.022 gm Insol
= Insol. 0.17

1/5 = 0.5 as $K_2Cr_2O_7$
= Fe 0.12

1/10 = 4.25 – 4.25 as $Na_2S_2O_3$
= Cu 2.32

1/10 = 4.6 – 4.7 as KCN
0.4 0.9 " $AgNO_3$
= Ni 1.38

$CaSO_4 + Na_2SO_4$ by diff.

4) Spec of 1st Press Cake

26.560	
11.383	17.4883

Moist 1.–

Took 12.965

1/1 = 0.587 gm Insol.
= Insol.

1/5 = 7.5 as $K_2Cr_2O_7$
= Fe

1/10 = 14.9 – 15.0 as $Na_2S_2O_3$
= Cu

1/10 = 10.3 – 10.1 as KCN
= 1.2 – 2.5 as $AgNO_3$
= Ni

[15]

5) Spec. of 1st Filtrate
3x3x0 cc.

Took 25 cc
91.950
62.917
= Sp. Gr.

1/5 = 0.3 cc $K_2Cr_2O_7$
= Fe
1/10 = 25 - 00 cc $Na_2S_2O_3$
= Cu
1/10 = 20.4 - 20.2 cc KCy
1.4 - 0.8 cc $AgNO_3$
= Ni

6) B.Y. Solution
By dissolving 1st Press Cake

Took 25 cc
81.332
49.058
= Spec.

1/10 = 4.7 - 4.8 cc $K_2Cr_2O_7$
= Fe
1/10 = 21.8 - 21.9 cc $Na_2S_2O_3$
= Cu :-
1/10 = 17.1 - 17.15 cc KCy
= 1.5 - 2.6 cc $AgNO_3$
= Ni

7) B.N. Washing Liquor
made by dissolving Copper Carbonate

Took 25 cc
86.933
53.660
= Spec.

1/10 = 0.9 cc $K_2Cr_2O_7$
= Fe
1/10 = 23.2 cc $Na_2S_2O_3$
= Cu
1/10 = 27.05 - 27.1 cc KCy
3.3 - 2.9 cc $AgNO_3$
= Zn.

8) Liquor from 2nd Treatment
#2 Press Cake

Took 25 cc
83.108
53.401
= Sp Gr.

1/5 = 0.1 cc $K_2Cr_2O_7$
= Fe
1/10 = 0.4 - 0.4 cc $Na_2S_2O_3$
= Cu
1/10 = 25.8 - 26.2 cc KCy
= 1.6 - 4.7 cc $AgNO$
= Zn

9) Press Cake from 2nd Treatment
#2 Press Cake

16.4295
12.3450 — 13.862

Insols

Insol 9.108 gms

1/1 = 0.05 gm Insol
= Insol.

1/10 = 11.0 = 0.95 cc $K_2Cr_2O_7$
= Fe

1/10 = 3.9 - 4.0 cc $Na_2S_2O_3$
Cu

1/10 = 14.9 - 14.8 cc KCy
3.5 2.0 cc $HgNO_3$

10) Insol. from #1 Press Cake
After Making B.V.

20.869
9.209 — 15.7810

Insol 8.457 gms

1/1 = 2.809 gm Insol.
Insol.

1/1 11.1 cc $K_2Cr_2O_7$
Fe

1/1 11.5 cc $Na_2S_2O_3$
= Cu

1/1 4.7 cc KCy
9.2 cc $HgNO_3$

11) Residue, after making B.V. titrating liquor

20.767
11.226

15.845

= Moisture

Took 13.427 grams

1/1 = 4.665 gm Insol.

Insol.
13.6 cc $K_2Cr_2O_7$

1/1 = Fe
10.5 cc $Na_2S_2O_3$

1/1 = Cu
10.8 cc KCy
2.6 cc $AgNO_3$

Ni

(2) M-Liquor from B.V. Meal
1750 cc

Took 25 cc
89.408
53.407
36.001 = Sp. Gr.

1/10 17.6 - 17.7 cc $K_2Cr_2O_7$

Fe

1/10 9.8 - 9.85 cc $Na_2S_2O_3$

Cu

1/10 28.3 - 28.2 cc KCy
1.7 - 1.8 cc $AgNO_3$

Ni

Restandardization of KCy soln.
(1 cc $AgNO_3$ = gm Ag)

1) 4.85 - KCy = 40.2 cc $AgNO_3$
2) 4.5 " = 37.3 " "
3) 4.5 " = 33.95 " "

1 cc KCy = gm Ag
1 cc KCy = gm Ni

13) Washings from B.V. Meal

Took 25 cc

41.048
55.878
— Sp. Gr.

1/10 = 11.8 - 11.85 cc $K_2Cr_2O_7$
— Fe

1/10 130 - 130 cc $NaVO_3$
= Cu

1/10 - 30.4 - 31.2 cc KCy
1.7 7.6 cc $AgNO_3$
Ni

Memo from Mr Pedersen
Ppt. from Floor Washings
50% Cu 50% Ni
3000 lbs per day
Cu Ppt. 2/3 Cu 1/3 Ni
3000* per day
Ni Ppt. 8/10 Ni 2/10 Cu
8000* ~ 10000* per day

14) Pure Nickel Sulphate Sol.

Took 25 cc -
89.830
61.150
— Sp. Gr.

1/10 = 21.9 - 19.7 cc KCy
16.0 - 1.4 cc $AgNO_3$
Ni

Based not detect Fe + Cu in 1 Liter of Sol

Sulph Acid used in dissolving Cu + Ni Carb. in #1 Tank

36.4418
34.6840
= 58.35 cc NaOH
= H_2SO_4

34.6840
34.4173
= 58.9
H_2SO_4

Sulph. Acid used in making B.V. Washing Sol. Said 470 cc

42.9029
41.6155
1.2873 43.75 cc NaOH
H_2SO_4 85.61 %

41.6155
39.9757
55.7 1.6398
85.69 %
H_2SO_4 85.57

15) Spec of Finished B.V. Meal
10/11/19

Nov. 12/19

Experiment to recover Nickel and Copper from Nickel Iron Mud from E.S.B.C. 110000# on Pile

Analysis of Sample

20.363	20.3620
15.678	17.5296
4.685	2.8334

Moisture :- 60.48 %

Dark 48.5072
40.7730
7.7342

All determination Wet Basis

1/1 = 0.0505 gm Insol HCl = Insol 0.65 %

Nickel Calculation :-

1/1 = 24.5 cc KCy, 20 cc $AgNO_3$ > Ni :- 1.808 %

Cu :- none

Fe faint trace

Acid Calculation

1/5 = 18.1 cc KCy, 2.8 cc $AgNO_3$ > Ni :- 6.57 %

1/5 - 0.37 cc Na_2S Cu :- 0.16 %

1/5 - 7.0 cc $K_2Cr_2O_7$ Fe :- 2.52 %

Analysis

	Water Sol	Acid Sol	Insol
Moisture 60.48 %	—	—	—
T. Cu 0.16	none	0.16	none
T. Ni 8.38	1.808	6.57	none
T. Fe 2.52	trace	0.52	none
Acid Insol 0.65			
Undetermined* 27.81			
Total 100.00			

* Note This is principally $CaCO_3$

Copper. Tried fusing to remove Molybdenum with a showing with hot cold and boiling water (100 grms + 200 cc water) — Broke up OK, but would not settle — Tried 100 grms Matte + 200 cc H_2O boiled and added H_2SO_4 until $K_4Fe(CN)_6$ test paper showed Fe in solution — This settles readily

Without Acid - boiled and allowed to stand 1 hr. Result :- Liquor turns up readily, but does not settle precipitate — Filtrate poured on top, and would not break up. — Slurry was dry the brown in color & stringy —

Contin.

With Acid :- Operation same as preceding but added ~~$K_4Fe(CN)_6$~~ H_2SO_4 until $K_4Fe(CN)_6$ showed Fe in sol. Result. Oper. 25 min. also produced settling had taken place to the extent of 2 1/4" in a total depth of 3 1/4" or 69.23% — no fresh on top — color a darker brown than preceding and more granular

Also tried drying matte and leaching with hot water (with and without acid) Results ~~Obtain~~ same as with wet matte.

As water sol. values are low & as acid must be used, & as results are no better than with wet, drying may be discontinued with.

One experiment was lost when almost finished, and by the time matter had been started, the matte had dried out appreciably, thereby a little higher in values than original.

Contin.

Details of Exp.

Took 100 grms Mud (wet) + 200 cc or grm. H_2O

Heat to boiling — Add H_2SO_4 until $K_4Fe(CN)_6$ showed sol. Fe —. Stirred until all lumps had been broken up — Added 100 cc extra H_2O to hasten settling.

(Note In practice would not break up every bit of lumps in Tank #1 but would use a strainer.)

Sulph acid added =

116.958
99.530
17.428 grms (85.69% H_2SO_4)
= 14.93 grms of act H_2SO_4

Before filtering Vol. = 350 cc — Washed with 20 cc Water

Vol. of Filtrate + Washings = 290 cc

Took 50 cc = 93.630
37.970
55.660 = Sp. Gr. 1.113

1/5 = 4.35 as H_2SO_4 = Cu 5.646 gm per l.
1/5 = 2.2 as $K_4Fe(CN)_6$ Fe 1.225 " " "
1/5 = 55.8 as KCy } Ni 31.857 " " "
2.2 as $AgNO_3$

(Contin)

Washed press cake with cold water until I judged it free of salines

Vol of Washings 140 cc

(Note In practice this may be cut down by using less water)

Took all for assay

			Sp. Gr.	
1/1 =	0.33	cc Hypo	Cu	0.0153 gpcc
1/1 =	0.60	cc $K_2Cr_2O_7$	Fe	0.0238 " "
1/1 =	24.9	cc KCy	Ni	0.988 " "
	5.0	cc $AgNO_3$		

These washings may be used instead of water for diluting original Mud.

49.810
26.859
22.951

Press Cake Wash

= 22.951 grams

19.708		19.748
12.254		17.525
7.454	Moist 22.31 %	1.983

Took 15.497 grms.

1/1 =	2.55	cc Hypo =	Cu	0.106 % Acet.
1/20	16.3	cc $K_2Cr_2O_7$ =	Fe	11.72 % Bass
1/1	4.2	cc KCy	Ni	0.144 %
	1.9	cc $AgNO_3$	Rest in Cake	

Contin

(Restandardization of KCy for this set
4.6 cc KCy = 33.6 cc $AgNO_3$

1 cc KCy = 0.02111 gram Ag
1 cc " = 0.00294 " Ni
1 cc $AgNO_3$ = 0.14 cc KCy Calc.)

These results calculated to lbs per Ton of Orig. Mud =

	1st Liquor	Washings	Wash Cake
Ni	184.76#	2.76#	0.49 %
Cu	32.74#	0.04#	0.66 %
Fe	6.70#	0.09#	53.80#

And when calculated to percent

	1st Liq	Washing	Wash Cake
Ni	98.27 %	1.46 %	0.27 %
Cu	97.90 "	1.19 "	0.91 "
Fe	11.06 "	0.12 "	88.82 "

Copies of letters to Mr. Mendenhall from Anaconda Copper Mining Co., Anaconda Montana signed by Fred. Larson Manager

The process proposed by Mr W. T. Burns Supt. of Electrolytic Dept.

Purifying Electrolyte at the Great Falls Electrolytic Copper Refinery

To maintain the impurities in the working electrolyte at a safe operating point an amount of sol. ~~[illegible]~~ sufficient to prevent building up of the impurities is daily drawn from the tank room to the purifying section. — With another of the present composition and a daily production of 280 Tons of cathodes, 6000 liters ~~tons~~ of sol. are drawn off each month.

The sol. to be treated is pumped directly to the building of concentrating tanks. Here, by the use of steam coils it is boiled down to 40 Be and is reduced to about 1/20 of its original volume.

Clarion.

From the concentrating tanks, the sol. is decanted to crystallizing tanks where it is allowed to remain for 4 days. During this time about 85% of the copper will have crystallized out as $CuSO_4 + 5H_2O$ — The M-liquor which remains, contains all of the free acid and impurities in a concentrated form, that were in the original solution & 15% of the copper. — This solution is removed from the tank by drawing a plug in the bottom. — After the blue stone crystals are well drained the plug is replaced and the tank is filled with water in which the blue stone is dissolved and returned to tank-room, as required.

The M-liquor from the crystallizing tank is of the following average composition:—

Contin.

	Grms per litre
Sp. Gr. 1.400	
Free acid	600
Cu	11
As	16
Sb	1
Fe	8
Ni	7

This acid is treated in the insol.-acide tanks where the Copper & Nickel of the arsenic and Antimony are removed leaving the Ni & Fe in solution.

The slime produced in the insol.-acide tank is of the following average composition.

Cu	43.0 %
SiO_2	0.4 "
Al_2O_3	0.4 "
CaO	1.0 "
Fe	1.5 "
S	3.0 "
As	21.0 "
Sb	2.5 "
Ni	0.5 "

Ag:- 25 oz. per Ton Au:- 0.1 oz. per Ton

Contin.

It is sent to the tunnelling for treatment.

The solution as it leaves the insol.-acide tank will have the following composition.

	Grms per litre
Sp. Gr. 1.400	
free acid	700.
Cu	trace
As	1.0
Sb	0.07
Fe	8.0
Ni	7.0

For the removal of the Fe & Ni the solution is again concentrated by boiling to 55° Be and decanted to crystallizing tanks, where the bulk of the Ni-Fe and salts of Lime & Magnesia present, drop out as basic sulphates.

The tunnelling solution, which the tanners call 'acid', is returned to the batch room as needed.

It has the following average analyses.

Contin.

	Grams per liter
Sp. Gr.	1.600
free acid	1100
Cu	trace
As	1.5
Sb	0.5
Ni	6.5
Zn	1.0

The slime remaining from the second concentration and sub-sequent crystallization is of the following average analysis

Manganese	4.24 %
Cu	1.04 %
Ni	11.90 %
Fe	12.80 %
free acid	14.85 %
Combined "	41.10 %

This slime is wasted by diluting with water and running into sewer. It is ~~likely~~ only product of the purifying process that is wasted.

In Sept. 1915 I shipped 1600# of this slime to the

Contin.

Lawton Copper Wks, where they attempted to treat it in their mixer plant for the recovery of the nickel. — Mr Cleaver reports that the nickel contents are too low to admit of its being profitably treated.

Approximately 75000# lbs per year, of this slime is reproduced.

[THIS BOOK WAS USED IN BOTH DIRECTIONS. THE FOLLOWING PAGES WERE FILMED FROM THE BACK END FORWARD.]

Sept. 25/19

Small Sple in tube from Mr Edison of Nickel Nitrate 9/25/19

Took 5.0 cc = 67.207 g
54.6070 Tare
7.7007 g = 0.8 cc Hypo sol

25 = Sple is 15% about
Cu is 0.0697%
or it is 1.04 grms per litre
Amt in the 30 cc's of Sple is 0.0312 grms
(Hence remainder out)

Sple in Small jar from Mr Edison
Marked Nickel Cut Tube
New Process
Not Separated 9/23/19

5.0 grms used = 23.6505 g
21.1883
2.4622 g PbCl2
= Pb is 33.63%

50 grms = 0.6 cc Hypo
= Cu is 0.072%

grms = { Pb is 1.6815 grms.
Cu is 0.0036 "

Have Took Sample

26

Sept. 26/19.

Sple of Nickel Flakes 1.992 grms
in Water glass

Given by Mr. Edison 11. AM. 9/26/19

1.992 grms — 21.2675
21.2390
0.0285 PbSO4
= Pb :- 0.99
1.992 gr 0.7 as Hydro
= Cu :- 0.23 %

(Sample given in mistake)
This sample all used up.

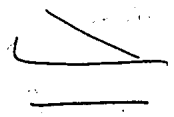
Sample of Nickel Flakes
given 1.00 P.M. 9/26/19
Correct Sample

1.987 grms = 19.4450
19.4250
.0200 PbSO4
= Pb :- 0.68 %
1.987 grms 0.7 as Hydro
= Cu :- 0.23 %

Sept. 26/19

Sple of Nickel Flake in Bottle
marked :- 4.987 grms

Took 2.0 grms =



PbSO4

= Pb :- Very slightly trace
as Hydro

2.0 gr — 1.0
= Cu :- 0.325 %

Actually in the 4.987 gr Cu :- ___ gm
Pb :- ___ "

Returned Remainder of Sple

Sept. 29

Spec on Water Glass from Mr Edison
Marked:- Residue in Nickel Bath
$NiSO_4$ Mg Acetate
This from 20 Moulds .005 1/2 thick each
* Water Sol consists of Nickel Sulphate
Acid Insol. Nickel Scales - Sand - and organic matter that shake with H_2SO_4 — Sawdust

Acid Sol. Ferric Iron 1/3
Ferrous " 1/3
Copper present
Manganese "
Lead none
Bismuth "
Phosphoric trace very faint
Aluminum " faint
CO_2 none
Arsenic considerable detect
As & Sb faint trace
CaO trace
MgO trace about 3 lbs in Mould

* No Water Sol. Cu or Fe Salts

Oct. 2 / 19

Problem for Mr Edison
Find how many lbs $NiSO_4 + 6H_2O$ and how many gals. water it will be necessary to use to form a saturated sol. at room temp. — Also how many lbs of Nickel Acetate must be added to form strength of 24 grams per liter

Took 500 gm $NiSO_4 + 6H_2O$ added 320 cc H_2O heated to about 100 °C with stirring — cool to 22 °C — Filter as dry as possible

Filtrate 430 cc @ 1.362 Spec @ 22 °C
Salt 238 grams

showing that in

	H_2O	$NiSO_4 + 6H_2O$
cc	320 gm or cc	500 gm
Liter	(60.15%) 744.2 gm	(24.85%) 493 "
gal.	2817 "	1866 "
10 gal.	12676.50 "	839700 "
10 "	= 2795.8 =	335 gal. 1852 lbs

Nickel Acetate for above 90.12 lbs to form strength of 24 grams per liter

[53]

Oct. 31/19

Metal from Mr. Dinwiddie 10/31/19
200 cc H_2O.
20 gm NaCl
5 cc HCl conc.
} Mixture used

Took Nickel plate from Cu plate forming a sol. X sponge

Sol to 300 cc Took 25 cc = 1/12
= 6.8 cc Hypo
= Cu 0.53 gram
1/12 − 1/6 = 2.0 cc KCy
= Ni 0.75

Sponge (Copper)
Took all, dissolve in HNO_3
1/10 = 13.4 cc Hypo
= Cu = 0.89 gm
Ni = trace (occlusion)

Oct. 31/19

Sponge contained precipitate from Mr. Dinwiddie 10/31/19
Ppt and Liquor both in beaker
Marked 800 cc H_2O 160 gm NaCl
6 plates stripped
15 mins required for each.

Filtrate shows trace of Chloride

Wt Ppt. Weighs :—
63.756
40.826 Total gram
22.930 Tare wt
63.756
55.003
8.753 wt used for assay

17.0 cc Hypo = Cu = 0.99 gm
4.9 cc KCy = Ni = 0.87 gm

To test :- Add HNO_3 until the X.S is known, and then add Nitric Concent until all Cu is ppt, as shown by KCy test and filter. Filtrate = Nitro. Pass Cl Gas Express Hydrate

Oct. 17, 9

Check on problem of Oct. 2 using $NiSO_4 + 6H_2O$ from Bol.

$NiSO_4 + 7H_2O$ + H_2O

Took 500 gms. + 300 cc

Heated to volume boiling, cool - filtered and obtained 410 cc sol. and 237 gms $NiSO_4 + 6H_2O$.

Therefore is

500 $NiSO_4 + 7H_2O$ 410 cc H_2O

237 300 cc

263 $NiSO_4 + 7H_2O$ 110 evap

Solution

410 cc = 300 cc H_2O 263 g $NiSO_4$

1 liter = 731.7 gms. 641.5 "

1 gal = 2769.5 " 2429.0 "

450 gal. = 1246275 " 1118600 "

450 gal = 2748# = 329.3 gal 2453.25

Spec. 1.68 @ 24°C

Check calculation for 7.6 [illegible]

$NiSO_4 + 6H_2O$ H_2O

262 : 110 :: 359575 : 150966

= 39.88 g

as a total %

95.
39.88
134.88 gals.

Oct. 8

Make Ferrous Carbonate for Mr. Edison

must be very finely divided and not show tendency to lump.

Dissolve $FeSO_4 + 7H_2O$ in water ppt $Fe(OH)_2$ with $BaCO_3$ — filter

I

~~Add Na_2CO_3 leaving excess Na_2CO_3 — boil — Settles readily~~

II

~~Add Na_2CO_3 using no excess $FeSO_4$ — boil — settles readily~~

I) $FeSO_4$ sol. + Na_2CO_3 sol in closed flask — xs Na_2CO_3 — Settled readily in cold — Washed several times by decantation, then in Lunatic flask — Mass hard to wash, so it turned out pretty fullers.

II) Made same as above except xs of $FeSO_4$ used — Washed same as above — Mass difficult to handle than #1 — Material of this character would varies filter by decantation.

III) Added xs Na_2CO_3 to $FeSO_4$ in closed flask — warmed slightly — allowed to settle once — tried to

(Contin)

Washed on vacuum filter — Material slimy and very hard to wash — Dried it and powdered under water, then filtered and washed — Washed more easily ~~than~~ but took on a reddish shade

(IV) Same as above; but persisted in washing on filter — Slimy — very hard to filter and wash.

(V) Added Fe_2SO_4 sol. to boiling sat. sol. Na_2CO_3 using xs Na_2CO_3 — ppt. dense and granular — Settled rapidly — filtered — Calcined washed and dried.

(VI) Portion of large batch — Added xs concen. sol. (warm) Na_2CO_3 to concen. sol (warm) of $FeSO_4$ — Settled and filtered — Filtered readily — Dried in hot air oven — Ground in water — Filtered and washed — Washed readily but seemed to contain large proportion of Fe_2O_3.

(VII) No sample

Took portion of above large batch before it was thoroughly dry — Tried a current of water

(Contin)

Cold, to see if it would disintegrate — As it acted as though it were coated or filled with oil, and did not disintegrate, experiment was discontinued.

(VIII) No sample

Added small amt. of H_2O_2 to neutral sol. (cold) of Fe_2SO_4 — Slimy ppt. not worth bothering with.

(IX) No sample

Same as above, but Fe_2SO_4 sol. was hot — Larger amt. of ppt. but slimy.

(X) No sample

Added xs Na_2CO_3 sol to Fe_2SO_4 sol heated — added H_2O_2 in very small portions — Then concentrated to Ferric Hydroxide.

Note In wrong column — Reason is really to form Fe_2O_3 in stead of $FeCO_3$ but to avoid the slimy a ppt. and see that resulting Fe_2O_3 shows tendency to agglomerate, and not to form fine powder

(XI) See following experiments

10/15/19

Sample of Electrolyte from Mr Dimmidale.

200 cc Soln representing 832 plates

Took 25 cc

65.438
39.157
26.281

Sp. Gr. 1.051

1/10 = 7.7 – 7.8 cc KOH

0.5 – 0.5 cc Hg No.

Ni :– 19.531 grms per litre

1/10 = 0.5 – 0.5 cc Hg No

= Cu :– 1.3 grms per litre

10/17/19

Soln of Nickel Nitrate Sol.

from Mr Edison 10/17/19

Entire amt. sol. in bottle = 110 cc

25 ccs = 72.577
44.797
27.780

Sp. Gr. 1.111

= 19.2973
19.2953
0.0020 Pb.

= PbO :– 0.0049 %

= " 0.0546 grms per litre

= " 0.006 grm in Soln

Oct. 17/19

Soln of Nickel - Copper Clippings in Cell Room Marked :–

Cl 1st Ox
Pb 10/17

Took all of Soln = 4.723 grms.

Could not detect any Pb Cl. after settling in ammonia + H_2S for 2 hrs and 48 hrs.

10/18/19

Sample of Cement Copper from Mr. Dimmidale 10/18/19

33.8283	33.8283
26.1575	31.0760
7.6708	2.7523

= Moist :– 35.88 %

Took 5 grms (Wet)

1/10 = 0.65 cc Hg No

= Nitric Sol Cu :– 0.09 %

= 13.5 cc Hg No

= Acetic Sol. Cu :– 1.81 % as CuO ?

1/10 = 22.5 cc Hg No

Metallic Cu :– 29.25 %

Balance is Metallic Nickel

10/20/19

Sample of Nickel Sol.
from New Dimmoddi
Marked 1.- 60 liters of sol.
after stripping 1280 plates

Took 25 cc 40.85 g
53.281
27.571

Sp. Gr. 1.103

1/10 = 0.6 cc Hypo
= Cu 1.- 1.56 grams per liter

1/10 = 11.3 cc Key
= Ni 29.48 grams per liter

✓

10/28/19

Sample of Nickel Flake
given by Mr Edison
Marked Flake sent from
Mr. Pedersen
Specific Grav Powders

Took
77.2743
75.3325
1.8 = 2 cc Hypo

= Cu 1.- 0.72 %
Pb none
by Chemical Test

✓

10/22/19

Sph of Nickel Electrolyte
from Pilot Exper.
from Mr Edison

Sp. Gr. 1.202 @ 28°C

Showed some Iron 1.3 at 1.
probably explains splitting
deposit

✓

11/7/19

3 Sph fused KOH from
L. Otts' Exp.

Top Color Green
2.5 grams = 58.7 cc $AgNO_3$
= KCl 1.- 4.93 %

Middle Color Brown
2.45 grams = 64.5 cc $AgNO_3$
= KCl 1.- 5.32 %

Bottom Color Almost Black
3.48 grams 4/10 = 27.2 cc $AgNO_3$
= KCl 1.- 3.95 %

Probably 1/2 of this is Iron raising to 7.90 %

10/25/19

Two Spls of Soda Electrolyte from Mr. O'Dair

(#1) Used for part 2 Mos. for testing 1/4" Tubes.

Their Analysis Sp. Gr. 1.175

NaOH	16.2 %	NaCl	0.02 %
T-Alkali as NaOH	16.5 %	Na_2SO_4	0.06 %
Na_2CO_3	0.4 %	Al + Fe	0.007 %

Took 25 cc

77.8665
48.7800
29.0865 = Sp. Gr. 1.163 (Room temp)

31.0 = 64.55 cc $N/10$ Sol. Phen. K 70.35 M.O.
= LiOH + NaOH = 16.19
Total Alkali as NaOH = 16.57 %
Na_2CO_3 = 0.51 %

1/5 = 19.5145
19.5119
.0026 AgCl =
= NaCl = 0.0182 %

3/10 = 0.0025 gm $BaSO_4$
= Na_2SO_4 0.017 %

10 cc = 0.002 gm $Fe_2O_3 + Al_2O_3$
= $Fe_2O_3 + Al_2O_3$ 0.027 %

(Continued)

Note Composition stated to be 15% NaOH + 11.2 gms LiOH per liter.

(#2) Spls from a cell

Their Analysis Sp. Gr. ——?

T-Alkali as NaOH	15.7 %	NaCl	0.07 %
LiOH + NaOH	14.97 %	Na_2SO_4	0.01 %
Na_2CO_3	1.00 %	Fe + Al	0.008 %

Spls full of sediment, hung on clean filter

Took 25 cc

77.7620
44.9462
28.8158 = Sp. Gr. 1.153 (Room temp)

3/10 = 43.5 cc $N/10$ Sol. Phen. K 44.65 M.O.
= LiOH + NaOH = 15.10 %
= Total Alkali as NaOH = 15.92 %
Na_2CO_3 = 1.09 %

1/5 = 19.2247
19.2186
.0061 AgCl
= NaCl = 0.0432 %

3/10 = 0.0023 gm $BaSO_4$
= Na_2SO_4 0.024 %

10 cc = 0.0014 gm $Fe_2O_3 + Al_2O_3$
= $Fe_2O_3 + Al_2O_3$ 0.019 %

10/31/19

Sample of Sediment from Primary Batteries

Spl from Mr Edison 10/31/19

26.9300	26.9300
24.6120	25.8180
2.3180	1.1120

= Moisture :- 47.97 %

90.2740
86.3570
3.9170 = 72.5 cc H_2SO_4 tot Phen

= NaOH 11.80 %
Na_2CO_3 trace

Residual Zn in above left 68.5 cc H_2SO_4 M_{10}.
= $Zn(OH)_2$:- 44.65 %

70.4572
52.4072
18.0500

1/2 = ____ gm $BaCO_3$
Na_2SO_4 trace

1/2 = 0.5 cc H_2SO_4.
= $(ZnO, NaOH)_2$ 0.20 %

3.917 gm = 0.08 cc H_2SO_4
= Cu 0.023 %
Na Cl faint trace

Mercury present
Trace of Potassium Iodide

See next page

Caustic

NaOH	11.80 %
Na_2CO_3	trace
NaCl	faint trace
Na_2SO_4	trace
Na_2S	"
$Zn(OH)_2$	44.65
ZnS	trace
$(ZnO, NaOH)_2$	0.20
Cu	0.013 %
Fe	trace
Hg	small amount
HgS	trace
H_2O	43.34 by difference
	100.00

33 % NaOH tot for 5, Phen

400 gm pure anhydrous NaOH + 1000 cc H_2O

18.3233
14.6900
3.6333 gm = 68.3 Phen cc H_2SO_4
69.3 MO

= J.a, as NaOH :- 29.20

Adjustment 990 gm tot + 186 gm H_2O

17.7805
13.5500
4.2305 = 66.85 Phen cc H_2SO_4
67.5 MO

NaOH 32.86 %

Said as 6K

Nov. 2/19

45% NaOH sol. for G Hart

546 gm pure NaOH + 786 gm H_2O

15.4117
13.5492
1.9625 = 36.5 Phen
37.10 Na2O as H_2SO_4

= J.R. as NaOH 40.82 %

Added 56 gm NaOH to above

16.4381
14.6910
1.7471 = 36.5 Phen
37.2 Na2O as H_2SO_4

= J.R. as NaOH 43.75 %

Added a little more NaOH (guess) on spatula.

15.8277
14.6910
1.1367 = 23.95 Phen
24.37 Na2O as H_2SO_4

= J.R. as NaOH 44.06 %

Hart said clear enough & used

Nov. 12/19

Cement Copper from Mr. Dimmadale

22.3010
15.6772
6.6238

22.3010
20.6250
1.676

Moist. 1- 25.16 %

Took 13.37 grms. (Lost)

3/10 - 1/10 = 22.3 - 22.35 as K_2Pt_6

= Cu 36.22 %

3/10 - 1/10 = as Kly

= Ni 30.31 %

~~Potas.~~ Nov. 14/19

Potash Extraction Exp. by Mr Edison from Feldspar, Examined Sol. only

#1 Marks Floated Feldspar Made 10/21/19 2 PM

1/10 = 21.0990
21.0950
.0050 Porcelain Error

= KCl 1- 0.0123 grms. in whole Sol.

#2 Marks Floated Feldspar $CaCl_2$ 10/31/19 2.25 PM

1/10 = 17.8500
17.7720
0.0780 Plat. Error

= KCl 1- 0.0861 grms in whole Sol.

Note Threw out Neutly sol. and saved residue for further investigation (21)

Nov. 12/19

Made up Sol. of Caustic Potash containing KCl and Iron in suspension (Ordinary Commercial)

10 cc = 32.1 cc H_2SO_4 Phen.
32.9 cc " MO
= J.A. as KOH 94.79 gr per l.
KOH
K_2CO_3

10 cc = ——— cc $AgNO_3$
= KCl 3.88 gr per l.

equiv. to 3.93 % KCl Caustic
or 39.3 % of Total Potash as KCl

(Take Silver Sulphate as 98% pure)
2 KCl 149.12 ; Ag_2SO_4 311.8 :: .398 : x =
100 cc Sol = 0.0826 grams Ag_2SO_4
Used 0.1000 grams.

1) 100 cc KOH Sol + 1.000 gr Ag_2SO_4
1/10 = 9.2 cc $AgNO_3$
= KCl 1.8 grams per liter.

2) 100 cc KOH sol + 100 cc H_2O + 1.000 gm Ag_2SO_4
1/10 = 7.0 cc $AgNO_3$
= KCl = 1.41 grams per liter

{1) 1/10 = 31.5 cc H_2SO_4 = J.A. as KOH 90.75 gm/l
{2) 1/10 = 32.2 cc " = " " 92.76 " "

Nov. 12/19

Made up a Solution of Comm'l K_2CO_3 and added KCl to it and filtered off insol. matter

10 cc = 15.5 cc H_2SO_4 Phen.
31.5 cc H_2SO_4 MO
= J.A. as K_2CO_3 108.96 grams per l.
Net K_2CO_3 107.52 " " "
KOH 1.44 " " "

10 cc 51.8 cc $AgNO_3$
= KCl 10.46 grams per l.

equiv. to 8.72 % of the J.-K. salts

1) 100 cc will take 2.19 gm Ag_2SO_4 (dry)
Added to 100 cc Sol 2.19 grams Ag_2SO_4 dry
shake up in flask
1/10 = 15.3 Phen. 31.15 MO cc H_2SO_4 = J.A. as K_2CO_3 107.76 g/l
1/10 = 5.6 cc $AgNO_3$ = KCl = 0.52 g/l
KCl eliminated 95.03 %

2) 100 cc Sol. + 2.38 grams $AgNO_3$ (dry)
1/10 15.15 Phen. 31.10 MO cc H_2SO_4. J.A. as K_2CO_3 107.59 g/l
1/10 5.6 cc $AgNO_3$ = KCl = 0.52 g/l.

KCl eliminated = 95.03 %

Nov. 17/19

Sample of Water from Storage Battery
Marked Tap Water, used for
Mixing Flake Wash (Wells)

Color :- slight opalescence.
Odor :- None
Sediment :- None

200 cc = 59.9260
59.9093 Dist Drip

T. Solids
59.9260
59.9216

Organic Mass :-

Cuestin

C25]

Nov. 19/19

Calculation for Mr. Edison

6 pc = Li_2O :- 4.00% = 6.41% LiOH
K_2O :- 8.00% = 9.53% KOH

make solution as follows
1200 cc 21% KOH contains
90 grms LiOH

Ratio in 6 pc 1 LiOH : KOH :: 1 : 1.48
" " Lepia LiOH : KOH :: 1 : 2.8
Therefore : To every 1200 cc of KOH made from 6 pc, (using LiOH as standard and assuming theoretical yields are obtained) 119 grms. of act. KOH from some other source must be added ✓

Tellurium Powder

Take cert-pure Tellurium powder - dissolved - free of Heavy Metals and Selenium and ppted by CO_2 or Metabisulphite - wash free of SO_4 Matter :-
Large particle 5 L 6 OK ✓
Small " 5 G. 4 pc

Nov. 20/19

1) Take 100 cc KOH sol (Dense)
+ 25 cc H_2O
+ .884 grm ($AgNO_3$)
Calculation that 1 liter =
J.a. as KOH 75.84 grm/l
KCl 3.104 " "

after adding $AgNO_3$ and shaking & filtering
Ohm Mg. g/l.
10 cc = 44.1 25.3 cc H_2SO_4 = J.a. as KOH 75.99
10 cc 6.5 cc $AgNO_3$ KCl :- 1.37
KCl eliminated :- 55.86 %

2) Took 100 cc KOH sol (Dense)
+ 25 cc K_2CO_3 " (Dense)
+ 25 cc H_2O
+ 1.479 grm ($AgNO_3$)
Calculated that 1 liter =
J.a. as KOH :- 78.32 grm/l
KCl :- 4.36 " /l.

After adding $AgNO_3$ & shaking & filtering
Ohm Mg. g/l.
10 cc 43.1 - 46.4 cc H_2SO_4 = J.a. as KOH 76.05
10 cc 5.3 cc $AgNO_3$ KCl 1.04
(CO_2 :- 22.93 grm per l.) KCl eliminated :- 75.46 %

Test for LiOH
NaOH
Al - Cu

Nov. 22/19 $Li_2SO_4 \times .4357 = LiOH$

Sample from Mr Edison, marked
Sample NaOH taken from drum
delivered Nov. 18/19
Standing in drum 24 hrs.
Sample given 11/22/19 (12 noon)
allowed to stand till noon 11/24/19
to allow drum to settle completely
Took clear portion for assay
Took 100 cc 189.880
72.907
116.973

Sp. Gr. 1.1697 @ Room temp

1/10 = 92.5 - 92.5 cc H_2SO_4 (Phen) 93.25 - 93.30 cc H_2SO_4 $N/10$

= Total as NaOH
Act NaOH
Na_2CO_3
KOH none

1/10 = 60.1541
59.9113
.2398
LiOH 10.05 gr/l
0.893 %

37.3278
37.0780
.2498
10.67
0.912

2/5 = 0.0028 gm Fe_2O_3 = Fe_2O_3
2/5 = 0.0004 " Al_2O_3 = Al_2O_3
2/5 = 0.0006 " CuO =

Caustic.

	I	II	Aver.
Total NaOH	16.38 %	16.39 %	16.39 %
Na_2CO_3	0.37	0.37 %	0.37 %
LiOH	0.893	0.912	0.903 %
LiOH + NaOH	16.16	16.11	16.11 %
~~LiOH~~	~~15.247~~	~~15.207~~	~~15.212~~ %
= gm/l.	10.45	10.67	10.56 %
Act NaOH	15.217	15.207	15.212
KOH	none	none	none

Fe_2O_3 " 0.006 %
Al_2O_3 " 0.00085 %
CuO " 0.0013 %
LiOH 1.848 gmm per 175 cc sol

15 %
30 %

Dec. 1/19

15% and 30% Nickel Sol. for G. Hack

Made up Rough Sol.

15.0 L 50

13.5525

= 14.4 gms of H_2SO_4

= Nickel 39.74 %

Took 1000 gm above sol + 300 gm H_2O

15.5560

13.5525

2.0035 P. 15.0 gm – 15.2 gm H_2SO_4

= Nickel :- 30.80 %

O.K.

Put ½ in bottle and labeled ½ plus equal wt. H_2O

Showed Mr. Hack 15% Sol

did Mr. Hack :-

15.3755

13.5515

1.8243 = 70 gm H_2SO_4

= Nickel :- 15.78 %

OK

Dec. 1/19

Sample of Nickel Electrolyte from Dir. Record Book 12/1/19

Tare 20 gm 77.470

51.700

25.770

Sp. Gr. 1.2885 Room temp.

1/1 = 2.4 gm Hypo

= Cu :- 0.78 grams per liter

Black ash : Ba S Assay

taken from 1 lb. bottle P.W. & R Co

30 gm = 24.4 gm H_2SO_4

= Ba S 79.72 % assay.

Figuring on 75% Average

Showed to Mr. Hack for approving Cu from above Sol. (Cu .78 g/l)

Cu		B-A ash
0.78 grams per liter	=	2.772 grams
1 gal = 3.785 lbs	=	10.492 "
400 gals.	=	4197. "
	=	9.25 lbs.

Probably need 15 lbs. Black ash in all

→ 0.02312 lbs. Black ash per gal of Electrolyte

(51)

Dec 2/19

Took 100 cc Nickel Electrolyte (bad) added xs $BaCO_3$ sol., so that all Cu was ppted. as shown by H_2S.
99% ~~Ppt~~ Ppt. settled in about 2 minutes. — Finely divided suspended ppt. easily returned by triple linen filter, but not quite by a single filter.
Ppt. after washing and drying, assayed:-
Took 0.619 gms.
1/1 = 0.42 gms $BaCO_3$
= $BaCO_3$:- 67.69 %
1/1 = 10.0 cc typo
= Cu :- 10.50 %
= CuS :- 15.79 %

$BaCO_3$	:-	67.69 %
CuS	:-	15.79 %
NiS	:-	16.52 % by diff
		100.00 %

Dec. 2/19 Mr Ewell. Reports 12/1/19

Formulas for Ni-Bath

$NiSO_4$	Distilled H_2O	Ni Acetate
474 lbs	95 gal.	45 #
237 "	47.5 "	22.5 "
118 "	31.66 "	15 "
118.5	23.75 "	11.25 "

For 78 Baths a strength 5.6 # fill lines and troughs in circuits & maintain circulation requires 260 gal. Where requires 123 # Ni acetate. — From time to time new sol. & compensate for evaporation the amt of water has not been definitely determined. Need in addition 50 # at the tank 123 # as above is possible for 78 new baths & 100 # per week thereafter until further notice.

Took 5 lbs Black Ash BaS, and 3 " Grey " for 260 gal of the Ni Sol. in N.R. Dept. 12/6/19
Removed Cu completely.

Dec. 8/19

Tellurous Acid

Reported that it is very unstable decomposing above 20°C = 68°Fah

breaks up into $TeO_2 \times H_2O$ ~~that~~ decomposed in that H_2O and only slightly sol. in cold H_2O

(Did not make it)

12/8/19

Prices for the Edison which is cheapest?

			lb
Peroxide of Hydrogen	3% @		13.3 ¢
or Barium Dioxide	90% @		23. ¢

H_2O_2 90% BaO_2

3% O :: 8.5% O :: 13.3 : x

x = 37.68 cent

or 1 lb. 90% BaO_2 on basis of active O is about 1/3 price of the H_2O_2

✓

17/9/19

Made following

1/1 gm	Stannic	Sulphate
1/1000 gr	Gold	Sulphate
1/1000 "	Platinum	"
1/1000 "	Palladium	"
1/1000 "	Thallium	"
1/10 "	Chromium	"

Sent from Wentlaff

* Ordered

Stannous Sulphate

Ppt Sn from $SnCl_4$ with NaOH — wash free of Cl — Dissolve in H_2SO_4 with x5 Sn and make x Ccl.

This should be Stannic Sulphate

Chromous Sulphate

17/9/19

22/10 Standardization of 1/10 MnO_4 Sol

1) 0.2232 gm Fe (99.9) = 0.2225 100 g = 40.2 cc
2) 0.2409 " " " = 0.2441 " = 44.25 "
3) 0.2281 " " " = 0.2274 " = 40.5 "

1) 1 cc = 0.005535 gm Fe
2) 1 cc = 0.005540 " "
3) 1 cc = 0.005600 " " ✓

Avge 1 cc = 0.005528 " Fe
0.00276 " FeO
0.00163 " MnO
0.006711 " $CuSO_4$

Dec. 10/19

Spec of Nickel Anode (drillings) from Mr. Bromley 4.30 Pm 12/9/19

Tare 2.007

1/10	27.45 - 26.95	cc KCy	
	10.0 - 5.6	cc $AgNO_3$	
	= Ni :- 78.13 - 78.40	—	78.27 %

1/1	53.✓	cc Hypo	Cu	17.3 %
1/1	8.1	cc $K_2Cr_2O_7$	Fe	%
1/1	0.000 g	gr Al_2O_3	Al	0.074

Do not accept fraction

This is Metal from S.B. Co after Cooling by Cadmium Belfast. N.Y.

Note — This must have been contaminated by Cadmium, Belfast.

Mr O'Dair claims that whoever cast them anodes, must have used a alloy that had previously been used for Copper

Analysis Nickel Anode 12/15/19

Lot of 340# for the Edison exp to

Nickel :-	98.40	98.50	98.40	98.30	98.60
Copper :-	0.60	0.10	0.10	0.50	0.40

✓

Dec 6/19

Took 100 cc Ni-Electrolyte — added 3 drops Peroxide Hydrogen — boiled and allowed to settle.

Result :-

2/3 Fe ppt. 1/3 in solution. free acetic permanent.

Reported Nicholls ✓

Took out 100 cc Ni-Electrolyte added 3 drops H_2O_2 — boiled then added freshly ppt. Ni(OH) ✓ Allowed to settle — Filter

Ppt		Filtrate	
	cc Hypo		cc Hypo
Cu	gr	Cu	gr
	cc $K_2Cr_2O_7$		cc $K_2Cr_2O_7$
Fe		Fe	
	gm Al_2O_3		gr Al_2O_3
Al		Al	

	Total	Ppt	Filtrate	Ppt	Fil
Cu	gr	gr	gm	%	%
Fe	gr	gr	"	"	"
Al	gr	gr	"	"	"

Added $MnCO_3$ to $FeSO_4$ solution

Ppted part of Fe by means of Na_2CO_3 sol — Separated from bulk of liquor — To liquor added Na_2CO_3 to neutralize reaction (showing acid Fe to be ppted) then added xs of $FeSO_4$ sol. — Filtered.

This gave 3 different fractions

1st	2nd	Mn-liquor
25.05 gr (Dry)	10.27 gr (Dry)	
Tank 3 gr	3 gr	1/4-7/5
7/10-1/5	1/10-1/5	

1 24.7 cc $K_2Cr_2O_7$ = Fe, 0.7 cc $KMnO_4$ = Mn
2 24.2 cc $K_2Cr_2O_7$ = Fe, 0.3 cc $KMnO_4$ = Mn
3 26.6 cc $K_2Cr_2O_7$ = Fe, 9.2 cc $KMnO_4$ = Mn

	Fe gm	Fe %	Mn gm	Mn %
1	13.76	~~13.94~~	0.113	39.37
2	3.37	14.11	0.024	8.36
3	1.48	7.95	0.150	52.27
	~~24.17~~ 19.61	100.00	0.287	100.00

Dec. 11/19

Spec of Mg Hydroxide ~~from Mr.~~ prepared for Mr Smith

Sample Mix 7 10/5/19

57.6850
55.3755
24.3095 = Phen 33.7 − MnO_2 34.6 MnO_4
= Tot. as NaOH = 18.54 %
act " = 17.38 %
Na_2CO_3 = 1.66 %

Spec returned to Mr Smith

Spec of 20% NaOH Sol. Assay for Mr Smith

Representing drums 6 & 7

20.0850
13.5503
6.5377 = Phen 32.85 − MnO_2 33.1 cc $N/10$ SO_4
Tot. as NaOH 19.69 %
act " 19.39 %
Na_2CO_3 0.42 %

Returned Spec. to Mr Smith

Examination of Copper metal furnished by Salem Lane Fe. 17/5/19

Cu — very faint trace

Dec. 12/19

Experiment in purifying Ni Electrolyte that had been treated by Ba(OH)2 in the R Dept.

510 Sple #1 Simply free of Cu ✓
Fe & Al present also CO_2 present. free acetic

✓ very poor separation

Sple #2 511 Cu free by Ba(OH)2
Fe and Al ppted by Ba(OH)2 ✓
did not boil off CO_2 or neutralize free acid

✓ Very good — promising

Sple #3 512 Same as #2 ✓
but excess Ni(OH)2 added and boiled

✓

Sple #4 511 Dup.
Same as #2 ✓ but boiled till free of O test by KI & starch

✓ No improvement by 2 boiling off O not necessary

Dec. 12/19

Sple #5 1518 Same Electrolyte
Evap. to 3/4 orig. vol. to remove part of free Acetic — Added H_2O to orig. vol.
— While boiling a sl. xs Ni(OH)3 added
Expected to oxidize Fe — ppt Fe & Al and neutralize free acetic

Sple #6 (1519) Same Electrolyte, Treated at Room temp. by oxidizing & decomp. Dioxogen and Ni(OH)2 ✓ in excess — Shook vigorously in bottle in filtered — Expected to oxidize Fe — ppt. Fe & Al and neutralize free Acetic

Sple #7 518 Tested by from Bdb. — Made pbl — Purified by boiling with Ba(OH)2 ✓ and Ni(OH)2 ✓ & allowed sediment to settle then filtered — No oxidizing agents used.
Sp. Gr. 1.297 @ 22 °C.

Showed pass on 1st deposit
0.1C. " 3 "
(Sediment brown on Ni-Anode)

Dec. 15/19

Sple #8 (519) Ni - Electrolytic

Cu ppts in Lump by BaO_2 - Filtered

Fe oxidized by BaO_2 very slowly and without

filtering excess of $Fe_2(OH)_6$ (fresh)

added to neutralize the free acid

— Did not neutralize completely

but almost —

No Fe went into sol.

Sp. Gr. 1,265

Very hard to filter

Gave poor deposit

Took some of 518

Supposed Cp. Nitro. Sol.

Recrystallized and dissolved x

x tals in Distilled water 5

Sp. Gr. 1,29 @ 20 °C.

Sple #9 and filtered

520 250 cc with nothing

added

First deposit poor

2nd good

Sple 10

521 250 cc + 1 cc 99.5 % Acid.

1st P. pretty good

2nd deposit particularly poor

* Crystals drained on filter pump and washed with cold water.

Dec 13/19

Restandardization of $KMnO_4$ Sol.

99.9 % 100. %

1) 0.2537 g Cu = 0.2535 29 1 = 38.9 cc Sol

2) 0.2535 " " = 0.25 2 2 1 = 38.65 "

3) 0.2572 " " = 0.2566 gm = 39.20 " "

1) 1 cc = 0.006536 gram Cu

2) 1 cc = 0.006527 " "

3) " = 0.006546 " "

Ave 1 cc = 0.006536 " "

Restandardization of KCy Sol.

	$AgNO_3$		KCy
1)	35.7	cc =	5.8 cc
2)		" =	"
3)		" =	"
1)	1 cc KCy =	0.02172	g Ag
2)	1 cc " =		" "
3)	1 cc " =		" "
Ave	1 cc " =	0.02172	" "
	1 cc " =	0.00200	" Ni

1 cc $AgNO_3$ = 0.134 cc KCy

Dec. 16/19

1) Sple of Drillings from Anodes
Marked :- Anodes direct from U.B. Co
Sple from Mr Bennett 17/12/19
This is before Melting by Cadmus

[illegible] grms used

1/1 =	[illegible]	cc $K_2Cr_2O_7$ =	Fe	1.33 %
1/1 =	0.4[illegible]	cc Hypo	Cu	0.1[illegible] %
1/10 =	35.1 - 35.[illegible]	cc KCl	Ni	98.0[illegible]
		cc $AgNO_3$	Ni	98.3[illegible]

Ave Ni :- 98.20%

(1cc KCl = 0.00589 grm Ni)

2) Sampling Nickel Sheet from Bloomfield Tomorrow
Rec'd from Mr Bennett 12/12/19

20.17 grms used :-
1/1
1/1

Dec. 16/19
Very small Sampling Sheet Nickel from Mr. Edisonay 17/12/19
Submitted by party who wishes to sell it

Sple 0.2[illegible] grms

1/1 -	cc Hypo -	Cu	
1/1 -	cc $K_2Cr_2O_7$ -	Fe	
1/1 -	cc KCl	Ni	
	cc $AgNO_3$		

Sample of Drillings from Nickel Anodes, Sple from Mr Clancy 17/12/19
Anodes before Shipment to Cadmus Bros.

Sple #1 20 grms - 0.5[illegible] cc Hypo
- Cu :- 0.18

Sple #2 0.6[illegible] cc Hypo
- Cu :- 0.21

Sple #3 0.6[illegible] cc Hypo
Cu :- 0.20

17/10/19

522 Mr Liquor from Sp. Nitro, X tals
Treated 1 cc 99.5% Acetic Acid

Sp. Gr.

(M.L. from 520 & 521)

1st Impure (Examine the tables)
2nd was much better.

---

523 Fresh X tallized NiSO.
250 cc + 1 cc 99.5% Acetic
1.265 Sp. Gr. at room temp

Perfect 1st deposit
but cracked after taking off.

---

524 250 cc. free from Leg Tunks
(treated) at D.R. Depo
+ 1 gm wet Ferric Borate Acetate

Poor at first
getting better gradually

17/10/19

525 250 cc Same Sol as 524
+ 2 gm wet $Fe_2(OH)_6$ + 5 drops Acetic

Poor at first ...

---

526 M-Liq from 521 - 523
Treated down and X tallized.
X tals treated on stand of
1.215 Sp. Gr. at room temp
+ 1 cc 99.5% Acetic

M-Liquor from 521 - 523 X tals
Contains:—

12/30/19

Tough Oleate of Lead
$Pb(C_{18}H_{33}O_2)_2$

Make in following :—
~~[illegible]~~ Hydrate Sol. of NaOH
~~[illegible]~~
+ Oleic Acid

Add to this sol. Lead Acetate

Dry ppt in air and then melt at 80°C, yr will form allow to cool.

12/30/19

Small portion of dark sediment which formed on the anode

Proved to be Iron
probably Ferric Iron Acetate

Jan. 7/20 {1 cc $AgNO_3$ = 0.00297 gr Ag}

Added $AgNO_3$ to the KCy Sol
and restandardized

	KCy		$AgNO_3$
1)	4.5 cc	=	27.7
2)	5.1 "	=	— lost
3)	4.05 "	=	24.5

1) 1 cc KCy = 0.01797 gr Ag
3) 0.01753 " "
Ave. 1 cc 0.01775 " "
× 0.27193 =
1 cc = 0.004827 " Ni

Two Spls of Pyrrhotite from Mr Edwin
Larger Spl initial marked 100

0.5 gm =	0.7	cc KCy =	Ni :-	0.19 %
0.5 " =	0.7	cc H_2PO =	Cu :-	0.26 %

(Small Spl in paper only 0.5 gr)

0.5 gr =	3.8	cc KCy =	Ni :-	3.19 %
0.5 " =	1.1	cc H_2PO =	Cu :-	1.44 %

		# per Ton		
Large Spl (Cream)	Ni	3.8	Cu	5.7
Small " Tails	Ni	63.8	Cu	28.8

Jan 6/20

Dettlefs formed on electrolysis
from Phem. Element Jan 1/6/20
Sp. Gr 1.261 @ 90°Fah.
105.614 gr Ni per liter
Mr. Edwin operations ish.

Schach Dettlefs which has been
KCy solution

My Sol.
25 cc 1/10 = 2.5 cc
53.0 cc KCy
6.9 cc $AgNO_3$ of Dettlefs Sol
= Ni :- 101.29 gm per l.
@

Found in a preliminary exp.
that a soln. sat. at 22°C
= Sp. Gr. 1.362
and contained :-
493 gm $NiSO_4$ + 6 H_2O per liter
= 105.93 " Ni " "

6

Jan 6/NO

Quick Method for estimating Ti in Pegmatite samples for combining Magnetic separation: For Heads-Tails & Middlings.

Tried out quite a few methods but found them either too slow or did not completely decompose the mineral.

Tried out the following:-

Time

Grind & Weigh 0.5 grms. - Add 1 gm $KClO_3$

+

Jan. 13/20

Samples of dilute Copper Sulphate sol. leached from CuO Discs made by G. Hart

Spec. from Mr Edison 1/13/20

Mark	Small Beaker	Large Beaker
~~Found to be contain~~		
Cu	Principal	Metal
SO_4	"	Acid
Reaction	Neutral	Neutral
Fe	none	none
Al	"	"
× Ni	faint trace	faint trace
Mn	trace	" "
Cl	"	trace
Alkali	present	present
Zinc :-	large amount	large amount

13 Just showed by Dimethylglyoxime

Copper Oxide used by G. Hart and tested by Mr L Edison Reagent

13 Zinc Oxide :- present

Jan. 13/20

Formula for making G. Hart's CuO discs

100 gram CuO
8 " $CuSO_4 + 1 Aq$.
6 cc 20% NaOH sol.

Found by actual calculation

$CuSO_4 + Aq + 2NaOH = Na_2SO_4 + Cu(OH)_2$
177.6 80

177.6 : 80 (100%)
40 (20%) : 8 : 18. g
as 20% NaOH sol = 1.226 Sp. Gr.
this 18. gm = 14.7 cc
instead of 6 cc

13

To use 40% NaOH for absolute precipitation

18 gram 20% NaOH = 9.0 gram 40% NaOH
40% NaOH sol = 1.438 Sp. Gr.
Therefore 9.0 gm = 6.25 cc

14

Jan. 13/19

Spec of KOH Sol. from Dem.
Spec sent from Mr Yeat 1/11/20
Assay for KOH, K_2CO_3, KCl, K_2SO_4
Fe - Al - Mn - S a SiO_2 ✓
also actual Potash

Took 25 cc 109.5575
73.1700
36.3895
= Sp. Gr. 1.456 Room temp.

1/10 = 287 - 28.63 cc H_2SO_4 Then
x 28.95 - 28.90 cc " MO
= J.A. as KOH 43.36 %
Act. KOH 42.57 %
K_2CO_3 0.97
2/10 = 29.0 cc $HgNO_3$ = KCl 0.81 %
1/10 - 1/10 = 16.8005
16.1210
K_2O + Cl 6795
= J - K_2O - 36.19 %

50 cc = 12.4073 | 12.4005 | 12.3970
12.3950 | 12.3950 | 12.3935
0.0073 | .0055 | 0.0035

16 ✓

SiO_2 0.0095 | $Fe_2O_3 + Al_2O_3$ 0.0072 ✓ | Fe_2O_3 0.0046
K_2SO_3 none | = Al_2O_3 0.0026
K_2S none | Mn_2O_3 none

Color :- Violet tinge
Sediment :- trace, black in color

Contin

Sp. Gr. at Room temp 1.456

KOH - K_2CO_3 + KCl as K_2O :- 36.91 %
Total act. K_2O :- 36.19 %

J.A. as KOH :- 43.36 %
Act. KOH :- 42.57 %
K_2CO_3 :- 0.97 %
KCl :- 0.81 %
K_2SO_4 :- none
K_2S :- none
Al_2O_3 :- 0.0026 %
Fe_2O_3 :- 0.0046 %
SiO_2 :- 0.0095 %
Mn_2O_3 :- none

Color :- Violet tinge
Sediment :- trace, black in color

Ash as Na2O 0.47

1.6 ✓

Jan. 6/20

Experiment to find approximate of various prices of operation etc. time element and yields in designing small plant to produce and keep on hand stock of double X tablets.

purified $NiSO_4$ Crystals from Laurence Nickel & Chem. Jar

1000. gram Crystal X tals, Vol = 780 cc
2.205 # = 0.0275 cu ft

Dissolved in distilled water, in large porcelain dish — and at boiling point added 15 cc Ba(OH)2 Sol.
=

Assay

Also added 320 gm Nickel Hydrate

Assay

Contin.

Acid before this treatment but neutral after. —

Just before filtering
Sp. Gr. 1.312 @ 88°C.
Vol. 1.675 cc

After filtering through linen — quite clear, small amt sediment passed at first.

~~Concentration 1.433 Sp. Gr. @ 90°C~~

Insol. = gr
cc

Concen. to 1.433 @ 90°C.
Cooled from 90°C to 72°C
Remet 505. gms X tals
850 cc Mother Liquor
1.243 Sp. Gr. @ 22°C

To the 505 gm X tals ~~added~~ 600 cc H2O added 200 cc H_2O, which reduced the total Vol. to 490 cc.

By the time Sol. had reached 70°C all salt was dissolved, showing a film on top. Film continued to stick even when heated to 100°C.

(Operation on next page)

Contin.

Boiled down to 360 cc total.
— then cooled from 110 °C to 28 °C

Result

380 gm 2nd Crystals
166 " " M-Liq
1.383 Af Lr. @ 28. °C

[ITEM(S) FOUND IN BOOK]

9/8/19

Mr. H_2SO_4 Solution

1.2083 gm Na_2CO_3 = 44.37 cc H_2SO_4 sol.
1.2072 " " 44.32 " "
1.2090 " " 44.42 " "
1 cc = 0.02732 gm Na_2CO_3
1 cc = 0.02716 " "
1 cc = 0.02717 " "
1 cc = 0.02722 " "
1 cc = 0.02518 " H_2SO_4
0.02055 " NaOH
0.02722 " Na_2CO_3
0.02881 " KOH
0.03459 " K_2CO_3

9/8/19

Mr NaOH Sol.

(1 cc H_2SO_4 sol = 0.02518 gm H_2SO_4)
36.8 cc H_2SO_4 = 26.8 cc NaOH
39.35 " " = 39.35 " "
35.90 " " = 35.87 " "
1 cc = 0.02518 gm H_2SO_4
1 cc = 0.02518 " "
1 cc = 0.02520 " "
1 cc = 0.02519 " "

[ITEM(S) FOUND IN BOOK]

Sept. 8/19

Potass. Iodide Solution for Cu

10 cc = .3 gm KI

150 gm to 500 cc

M.10 Hyposulphite Soda 9/8/19

1) 0.2506 gm Cu 99.9% = .2503 100% = 38.6 cc sol

2) 0.2040 " " " = 0.2038 31.5 " "

3) 0.2117 " " " = 0.2115 32.65 " "

1) 1 cc = 0.006486 gms Cu

2) 1 cc = 0.006470 " "

3) 1 cc = 0.006519 " "

Ave. 1 cc = 0.006491 " Cu

M.10 $K_2Cr_2O_7$ Solution 9/8/19

1) 0.2148 gm Fe 99.7 = 0.2142 (100) = 38.95 cc

2) 0.2159 " " " 0.2157 " 38.9 cc

3) 0.2345 " " " 0.2341 " 41.95

1) 1 cc = 0.005528

2) 1 cc = 0.005560 gm Fe

3) 1 cc = 0.005580 " "

Ave 1 cc = 0.005570 " "

0.00796 " Fe_2O_3

Potass. Iodide Solution for Ni

10 cc = 1. gm KI or (10%)

[ITEM(S) FOUND IN BOOK]

9/10/19

Check on my H_2SO_4 Sol.
from Silver Lake
Their figure = .997 % my

1) 2.004 gm Na_2CO_3 = 36.77 cc Sol
2) 2.002 " " = 36.18 cc "

3)
1. 1 cc = 0.05450 gm Na_2CO_3
2 1 cc = 0.05452 " "
Aver. 1 cc = 0.05451
Standardized at 22 °C

1.0285 Normal
Multiply readings × .972

Check against my NaOH
1 cc = 0.02519 gms H_2SO_4

NaOH my		H_2SO_4 my
39.0 cc	=	19.50
47.12 cc	=	23.56

1) 1 cc = 0.05064 gm H_2SO_4
2) 1 cc = 0.05051 " "
Aver. 1 cc = 0.05058 " "
or 1.03 Normal

Multiply readings by 0.9703 for my
practically [illegible]

[ITEM(S) FOUND IN BOOK]

Sept. 10

$AgNO_3$ Sol.

1) 0.0806 gm KCl = 40.1 cc $AgNO_3$
2) 0.0859 " " = 42.5 " "
3) 0.8068 1/10 " " = 39.8 " "

1) 1 cc = 0.00200 gm KCl
2) " = 0.00202
3) " = 0.00201
Aver " = 0.00201 " "
" " = 0.00149 " Ag

Sept. 12

KCy Sol
(42 gm to 2 liter)
(1 cc $AgNO_3$ Sol = 0.00149 gm Ag)

	$AgNO_3$	=	KCN
1)	31.7		40.0
2)	37.9		46.8
3)	35.35		44.7

Aver 1 cc KCy Sol = 0.02312 gm Ag
2) 1 cc " " 0.02365 " "
3) 1 cc " " 0.02309 " "
Aver 1 cc " " 0.02312 " "
" " " 0.006287 " Mi

[ITEM(S) FOUND IN BOOK]

10-24-19

Mr Edison

This is a recent sample of nickel flake made by plating on lead cathode.

We made a test for loading & got poor feeding.

I would also like to get a check analysis for lead

Dave

Dougherty

Test for Lead

Note Color of Reg flake & Chem flake

[ITEM(S) FOUND IN BOOK]

Doughery

Here is Samples of fused KOH which is said to Contain 2% KCl — It was fused to thin liquid then fire withdrawn + hot ashes covered it it required 4 Hours before it Congealed Samples are from Bottom middle + Top — please assay quantatively for Chlorine —

[ITEM(S) FOUND IN BOOK]

Cu 23%

Equivalent H_2SO_4 37%

Impurities Ni .4%

Fe .3%

Arsenic .15%

Free H_2SO_4 1%

[ITEM(S) FOUND IN BOOK]

Cu 23%

equiv Hydr. 37%

Impurities and Fe

0.3 Fe

0.1 As

1.00 per Ht.

[ITEM(S) FOUND IN BOOK]

Doughearty 10-25-19
This refers to the
bottles of Electrolyte

Mr. Edison:

Herewith are two samples of Soda electrolyte which we have used during past two months for testing 1/4" tubes.

The composition of electrolyte should be 15% NaOH + 11.2 grams LiOH per liter.

Our analysis on Sample #1 was: NaOH = 16.2 ; Acc. = 16.5
Gravity = 1.175 NaCl = .02 $NaCO_3$ = .4
$NaSO_4$ = .06 Al+Fe = .007

Analysis of sample #2 which was taken from a cell is NaOH+LiOH = 14.97 $NaCO_3$ = 1.00
Alkalinity = 15.7 NaCl = .07
$NaSO_4$ = .01 Fe+Al = .008

I would like to get a check on this

[ITEM(S) FOUND IN BOOK]

Mr. Edison :-

Here is about what is meant, relative to Potash Sol.

All Potash KOH - K_2CO_3 - KCl is calculated to K_2O —

45% K_2O is guaranteed, which insures against weak solution.

They will charge - 23½ cents per lb. for 90% of the K_2O contents.

Illustration :-

100 lbs. Sol. × .45% = 45 # K_2O

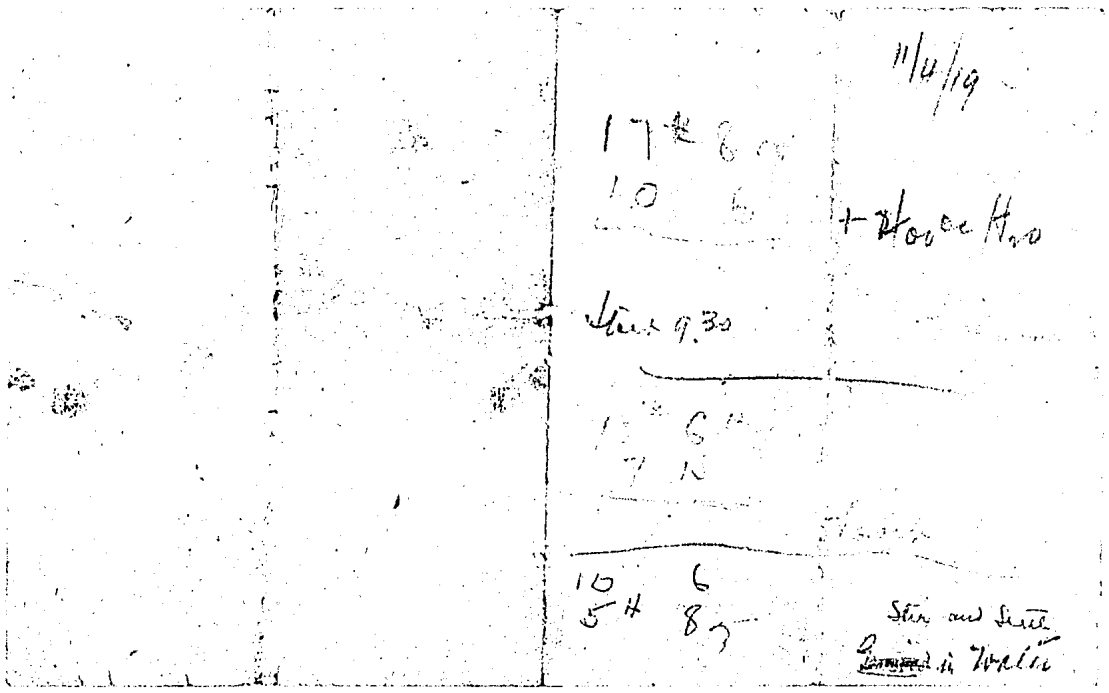
(45 # K_2O = 53.6 # KOH)

45 # K_2O × .90% = 40.5 # K_2O charged for

40.5 # K_2O × 23½ cents = $9.52

100 # of 53.6 % KOH = $9.50

[ITEM(S) FOUND IN BOOK]



[ITEM(S) FOUND IN BOOK]

Nov. 24/19

Mr. Edison:-

Please find herewith final 4 samples of Iron Oxide.

If they reduce as easily as samples #1 and #2, and the product is satisfactory, I will figure on Factory operation. I believe all the four samples will be found to be denser than #1 and #2.

Made as follows:-

Used Copperas and Soda Ash from Silver Lake. — By actual test I found that 4 lbs. Soda-Ash would precipitate the Iron from 10 lbs Copperas, allowing at the same time a slight excess Fe to 1. — Copperas & Soda Ash pulverized and Mixed Dry. —

(2)

Sample #3:- Placed in shallow pan, moistened slightly with water, and heated to 150° C. — ~~[illegible]~~

Product Red and granular. — Covered completely 14 times with boiling hot water, allowed to stand a few minutes, and drained by means of Suction. — Dried on hot plate.

Sample #4 Same as #3, only washing was accomplished with hot water, by agitation, ~~[illegible]~~ settling, and decantation.

Sample #5 Dry Mixture of powdered Copperas and Soda Ash added directly to cold water. — Transferred to percolator and

[ITEM(S) FOUND IN BOOK]

(3)

a gentle current of cold water from faucet allowed to pass from bottom, up through mass, at top. I did not measure amount of wash water necessary for complete washing but it was not very much.

Every particle was kept in motion, whereas a flame on the opposite direction might have caused packing. — Finished on Sulzer filter and dried ~~on [illegible]~~ on brass pan directly over gas flame to minimum temperature of 212°F.

Sample #6 same as #5, only washing by agitation with hot water, settling and decantation. Finished same as #5.

E. E. Ringling

[ITEM(S) FOUND IN BOOK]

Jan. 12/20

Mr. Edison:-

Mr. Merrill of Storage Battery Plant has called my attention to, and furnished samples of two products that they have a market for, but which is not very satisfactory.

A superficial examination of them leads me to believe that we could utilize them to greater advantage in business, and just as soon as possible I will outline in detail what I have in mind.

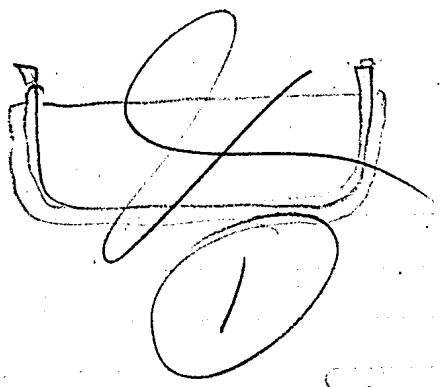
The first in importance is Cement Copper of which they are producing 1800* per week (wet). This contains metallic Nickel, and if we decide to work up ammonias, we could also utilize the Cement Copper Mr. Dinwiddie is producing at his stripping-plant.

The other is a mixture of Metallic Copper Cement, Cuprous Chloride, Gypsum etc. which accumulates in bottom of tanks. They turn out about 1000* per week (wet).

E. Ashcroft

Arrange plant to take care of these also & Dinwiddies stuff in addition to Mud & Z Crystallization Mass of

[ITEM(S) FOUND IN BOOK]



1

Evaporating pans wood lead lined with lead Coils - for Evaporating down for 1st Crystalizations. —

Enamelled Steam heated dissolving - tank, from this pumped to Enamelled Evap pans — 3rd Crystn measures approximates

2

Tank for holding mother liquors.
What shall we do with this —

Small enamelled tank Steam heated for precipitating MgOH. boiling — Washing by decantation till free of Sulphates, then small filter press to get Cake. This used for Neutralizing plating baths + about 300 lbs per week —

[ITEM(S) FOUND IN BOOK]

(3)

Mud Lay Out

Layout for Cement Copper
on the CuCl residue

[ITEM(S) FOUND IN BOOK]

To make a Keep on hand
Say 4000 lbs NiSO4
Double Crystalized.

1 Enamelled Kettle
steam jacketed —

One tank enameled with
stirrer to break up hot
Crystals,

1 Centrifugal to
clear Crystals of
Mother Liquor

Tank for Mother
liquor —
~~Sat Letter~~ How about
filtering

[ITEM(S) FOUND IN BOOK]

Alcohol Sol

Mg Sulfate	Soluble
Ni Sulfate 6 aq	Sol
Ni " 7 aq	Very Sol
Chromic Soy 5 aq	Very Sol
Chromous Sulfate	Slightly
Cobaltous Sulfate	2.5 in 100 al
Vanadium Soy	Soluble
Zinc Soy	Slightly
Uranyl Soy	4 pts to 100

250 cc Cu free Ni Soy

2 gms	Ni acetate	OK	7 lb run
1 "	acetic acid	OK	7 lb "
5 "	"	OK	11 lb "
—	nothing —	OK	7 lb "
2 gms	Ferric acetate	OK	5 lb "
2 "	Ferric Sulfate	OK	7 lb "
4 "	"	Bad after	22 "
4 "	Ferric acetate	OK	4
1 "	Cerous Soy	OK	1st Runs
½ gm	$Al_2(Soy)_3$	Bad after	11 "
1 gm	Manganous Soy	OK	9 lb "
1 gm	Cd Soy	Bad after	5 lb "

[ITEM(S) FOUND IN BOOK]

good

Silver — 3
Hg2 SO4 4
Cobalto SO4 2
4 Ferric Acetate 4

Bad —

glucose,
8/4
1 CuSO4 —
1 Zn SO4
1 Cd SO4
1/2 Alumina SO4
Ammonia SO4

Rather

[ITEM(S) FOUND IN BOOK]

Big bath Cu only out OK		10	Runs
" Cu 7½ al. out by BaO_2		6	
" " " neutralized by NiO_4		7	
CP Nickel (Daughtery)		4	
Big Crock Cu out only —	1 gm acetic	6	
" "	5 "	11	
" "	nothing changed	7	
" "	2 ferric acetate	5	
" "	2 " SO_4	7	
" "	4 ferric acetate	4	
" "	$CuSO_4$	Bad after	30 Runs
" "	$ZnSO_4$	Bad after	29 "
" "	Chromic Sulfate	2 Runs	
" "	½ Aluminum Sulfate	Bad after	22 "
" "	$MnSO_4$	9	
" "	$CdSO_4$	No peaks	

Big

Big Crock Cu out —	BaO_2 treated	6
513 Daughtery		5
514 "		6
Big Crock Cu Out —	3 mlg Mercurous SO_4	4
"	5 " $AgSO_4$	3
"	1 gm Glucose	Bad
"	10 cc 8/4	Bad

[ITEM(S) FOUND IN BOOK]

Dumortierite -
Cement Cop
Cu Cly
Muscovite -

2nd Crystalline -
Decolorized Cu *

[ITEM(S) FOUND IN BOOK]

Total 45%

546 Total 786 H.O

15.4117

13.5497

1.8625

36.5 Phenol

37.0 H.O

Total 40.97 %

Added Copper 15 ml

16.4745

14.6910

1.7475

36.5 Phenol

37.2 H.O

Total 43.75 %

15.8277

14.6910

1.1367

39.5 Phenol

24.87

Total 44.06 %

(tin)

10% Copper = 46.00

4% Tin = 3.00

[ITEM(S) FOUND IN BOOK]

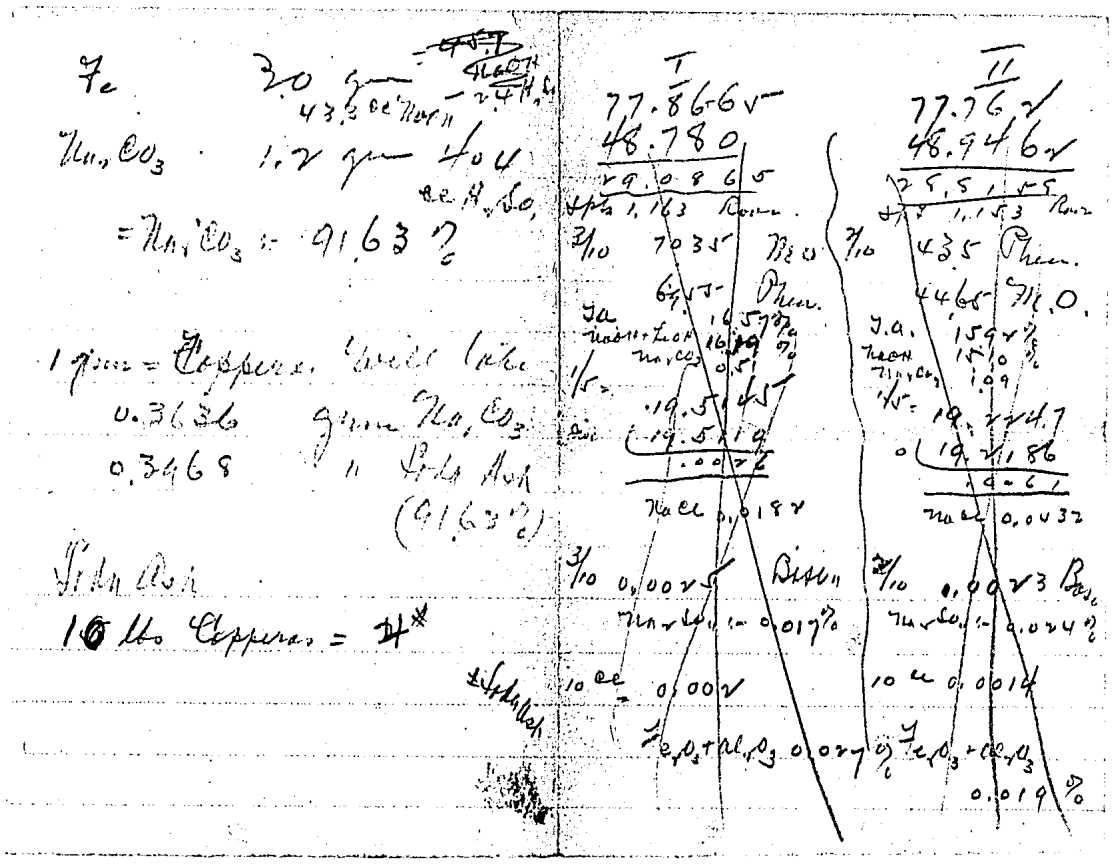
10/29/97

Item sent from
the Pathology
Specimen Processing

97-97-43
75,332 Turf
1.8 = 2 [illegible]
= Cu 0.72 %

Pb [illegible] none

[ITEM(S) FOUND IN BOOK]



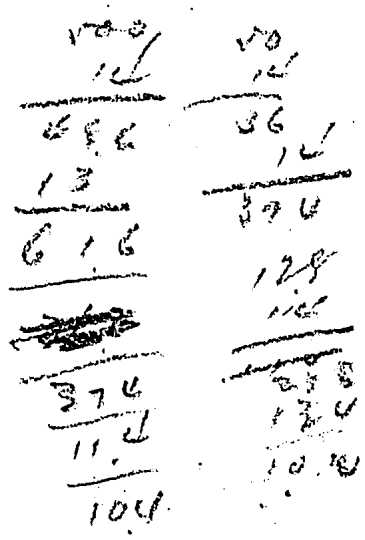
[ITEM(S) FOUND IN BOOK]

11.8
.62
5.6
2) 4.83
3) 4.87
30.1 Hg/Kg
29.1
30.1

165
1.8
0.83

21.5 372
165 2188
4.87 5.56

[ITEM(S) FOUND IN BOOK]



[ITEM(S) FOUND IN BOOK]

Water	6.4 #	
Ni + Cu Carbonate	40.48 #	2500 # = 61.7 cu. ft.
Ni "	39.87 #	
Cu "	34.92 #	
H_2SO_4 (61.66°)	114.47 #	

Found 1 # Mixed Carbonates will take up 93 %
2500 # Mixed Carbonates = 375 # # H_2SO_4 (93 %) H_2SO_4

Tank 4' x 6' will hold 75.36 cu. ft. Mixture at left
" 6 x 10 " " 226.24 " " @ 8'
or over 3 days supply

[ITEM(S) FOUND IN BOOK]

#1 Very small amt Mich Flow
2 Did not clear away
3 Concentrate
4 Add little Mich Flow

B.V. Work Up
2620 cc
Took 200 for Apt
Insol, from above
450 grms.

#1 x #4 good dark color
x Cleared #3 x 3 Clear

Filtrate
1-3-4
Stored 3 supernatants
part of each 3 open standing

Boogy + 320
Apt 1 Insol
Sol
Sol 30 cc
Insol

1 x S $MnCO_3$
2 x S $FeCO_3$
Both settle readily, but
7S $MnCO_3$ settles more
readily

3 Add $MnCO_3$ + $FeCO_3$
in acid — filter
Wash free of Sulphate

4 Add $MnCO_3$ + $FeCO_3$
in acid — Settle, ~~fol~~
decant once, Filter, dry —
Pulverize and Weigh

1st Eluate
16# 6 g
4+ 8

B.V. Work Up
~~1st Eluate~~, 1750 cc
of M-Liquor from B.V. Work
15, 8 g
4 8
x-100 gms
Wash liquor for B.V. x Cu
900 cc

[ITEM(S) FOUND IN BOOK]

Potass. Cyanide

24.35 gm per litre

KCN

4.903 gm per litre

1903

Hydro

24.8 gm per litre

Nitric Sec

1.4 gm per litre

Hydro Sec

1.07 gm per litre

50 gm KI per 500 cc

50 " I " 500 cc

Ag NO3 Sec

16909 47 gm Ag NO3 to litre

9.44